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Ice formation modes during flow freezing in a small cylindrical channel

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ABSTRACT

Freezing of water flowing through a small channel can be used as a nonintrusive flow control mechanism for microfluidic devices. However, such ice valves have longer response times compared to conventional microvalves. To control and reduce the response time, it is crucial to understand the factors that affect the flow freezing process inside the channel. This study investigates freezing in pressure-driven water flow through a glass channel of 500 μm inner diameter using measurements of external channel wall temperature and flow rate synchronized with high-speed visualization. The effect of flow rate on the freezing process is investigated in terms of the external wall temperature, the growth duration of different ice modes, and the channel closing time. Freezing initiates as a thin layer of ice dendrites that grows along the inner wall and partially blocks the channel, followed by the formation and inward growth of a solid annular ice layer that leads to complete flow blockage and ultimate channel closure. A simplified analytical model is developed to determine the factors that govern the annular ice growth, and hence the channel closing time. For a given channel, the model predicts that the annular ice growth is driven purely by conduction due to the temperature difference between the outer channel wall and the equilibrium ice-water interface. The flow rate affects the initial temperature difference, and thereby has an indirect effect on the annular ice growth. Higher flow rates require a lower wall temperature to initiate ice nucleation and result in faster annular ice growth (and shorter closing times) than at lower flow rates. This study provides new insights into the freezing process in small channels and identifies the key factors governing the channel closing time at these small length scales commonly encountered in microfluidic ice valve applications.

1. Introduction

Microfluidic systems have been widely employed for chemical and biomedical analysis as they enable high analytical throughput at low sample volumes as well as ease of integration at low cost [1–5]. However, miniaturization and commercialization of fully integrated microfluidic systems have been hindered by the lack of reliable flow control elements [6]. Flow control is typically achieved through conventional microvalves, which tend to be miniaturized versions of their macroscale counterparts. They are generally actuated through contact between a micromachined orifice and a flexible membrane [7]. Thermal [8,9] or piezoelectric [10,11] actuation deforms the membrane, which makes contact with the valve seat to close the channel. Such microvalves are difficult to fabricate, suffer from unavoidable flow leakage due to the presence of moving elements, and the small gap between the sealing element and the valve seat can generate excessive flow resistance [12].

Considerable effort has been directed at developing innovative microvalves that have alternative actuation mechanisms using phase-change materials including hydrogels [13,14], sol-gels [15], paraffins [16,17], and ice [7,18–22]. This alternative microvalving approach introduces other challenges while benefiting from operation with no moving parts. For instance, all the microvalves, except the hydrogel and the ice-based phase change valves, require an external sensor for timing the valve closure. In hydrogel and paraffin valves, the phase change material is in direct contact with the working fluid, raising contamination and biocompatibility concerns [21]. In addition, accurate positioning of the molten paraffin at the desired location, as required for proper sealing, is difficult [17]. Moreover, once the paraffin solidifies and plugs the channel, an external pressure is required to re-open the valve, thus making it more suitable for a 'one-shot' operation. In contrast, ice valves utilize the working fluid itself as the phase change material to shut off the flow; they are non-invasive and leak-proof, have no moving parts and no dead volume, are free from foreign material, offer no

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Nomenclature

A	channel cross-sectional area
c_p	specific heat at constant pressure
D	channel inner diameter ($D = 2R$)
h	convective heat transfer coefficient
k	thermal conductivity
l	channel length
L	latent heat of fusion
$\dot{m}$	mass flow rate
n	refractive index
Nu	Nusselt number ($Nu = \frac{hD}{k}$)
P	pressure
$\dot{Q}$	heat transfer rate
r	radial coordinate
R	channel inner radius
ΔR	change in radius during single time step
Re	Reynolds number ($Re = \frac{\dot{m}D}{\mu A}$)
t	time
Δt	time step
T	temperature
ΔT	temperature difference between ice-water interface and the external channel wall ($\Delta T = T_i - T_{wall,ext}$)
W	water flow rate ($W = \dot{m}/\rho_l$)
x	transverse position relative to channel centerline (Fig. 4)
y	vertical position relative to channel centerline (Fig. 4)
z	axial or streamwise position along the channel centerline (Fig. 4)
Δz	length of a unit control volume in the axial direction

Greek symbols

μ	dynamic viscosity
ρ	density
θ	angle between the normal and incident/refracted light rays

Subscripts

ann	annular region
bs	borosilicate glass
cav	test cell cavity
cond	conducted heat energy
conv	convected heat energy
ext	external
f	freezing
i	ice-water interface
ice	ice
in	inlet
int	internal
l	liquid phase (water)
lat	latent heat
m	mean
n	nucleation
o	initial
out	outlet
open	open area of channel
rel	released (energy)
s	solid phase (ice)
sens	sensible (energy)
wall	channel wall
water	water

Superscripts

i	index for a unit control volume
j	index for time step

extra flow resistance in the default open state, and are easy to fabricate at low cost. As a result, ice valves have been explored for applications ranging from lab-on-a-chip devices where the rate of reaction can be controlled by freezing the flow of reactants [7,21] to complex microfluidic systems involving separating and routing fluids [23] by freezing small water droplets that are immiscible with fluids. However, ice valves have a major drawback that they have a long response time on the order of seconds (~2.5–30 s [18–21]) compared to a few milliseconds (as low as 1 ms [24]) for the conventional microvalves. The long response time is principally due to: (1) the high thermal inertia and limited cooling capacity of the refrigeration systems used to cool the liquid, and (2) the need to supercool the working fluid to temperatures below the freezing point to trigger phase change. To speed up the cooling process, multi-stage thermoelectric cooling units have been employed that generate a large temperature difference between the cooling surface and the channel [22], or the initial temperature of the system has been lowered by pre-cooling the fluid/device. Even with these advances, the response time of current state-of-the-art ice valves remains on the order of 1 s [22].

While most prior studies have focused on design modifications at the device scale or changes in operating conditions, the ice formation and growth process occurring within the channel flow remains relatively unexplored. Once nucleation occurs, water-to-ice conversion takes a finite amount of time [25], which may constitute a considerable duration of the valve response time, depending on the mode of ice formation. The current study focuses on obtaining a more complete understanding of the fundamental flow freezing process occurring inside small cooled channels as a step toward arriving at techniques to reduce the channel closing time.

Several studies [26–38] have explored the different ice formation modes and the thermophysical parameters affecting their growth behavior in supercooled, large-diameter channels under different flow configurations, including stagnant liquid, flow entering an empty channel, and continuous flow through the channel. The temperature history during the freezing of pure water in a cylindrical channel is introduced in Fig. 1 for the canonical case of stagnant conditions [26]. The channel is initially placed into a cold isothermal environment well below the equilibrium freezing point. When the water is cooled, it does not necessarily freeze at the saturation freezing point (T_f = 0 °C at atmospheric pressure), but must be cooled by several degrees below T_f before nucleation occurs [27], *i.e.*, the nucleation temperature $T_n < T_f$. For example, the nucleation temperature for normal tap water is 4–6 °C below the freezing point [28]. Water existing in liquid phase below the equilibrium freezing point is in a thermodynamically metastable state and is classically termed as undercooled or supercooled water, with the degree of supercooling given by $(T_n - T_f)$. For microchannel closure, the phenomenon of supercooling is important in two ways. First, the time required to achieve supercooling can be long. For example, in a microchannel (233 μm × 172 μm × 38.2 μm) water had to be supercooled to −17 °C before the initiation of nucleation, which required a time of ~3–8 s [21]. Second, in supercooled water, ice does not grow as a solid layer, but rather as dendrites which can have a markedly faster growth rate and hence affect the channel closing time. As shown in